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EVALUATION OF A NON-CATALYTIC COATING FOR METALLIC TPS

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ABSTRACT

A commercially available ceramic coating has been evaluated for application to metallic heat shields for Shuttle-type entry vehicles. Coated Inconel 617 specimens were subjected to thermal shock cycles, surface emittances were measured, and surface equilibrium temperatures were measured for coated and oxidized specimens exposed to an arc-tunnel environment. The coating adhered very well to the metal and appeared to be very non-catalytic.

INTRODUCTION

For the same earth entry conditions, a metallic surface of an entry vehicle will usually be subjected to a higher heating rate than a non-metallic surface. This difference occurs because metallic surfaces are generally catalytic to the recombination of dissociated air molecules, and the energy of dissociation, released during recombination, adds to the heat load.

Several advanced thermal protection systems are being considered for future Shuttle-type entry vehicles (ref. 1). One concept being considered for the 1600-2000°F range has an Inconel 617 outer face sheet (ref. 2). To prevent this surface from being exposed to the added heat load due to recombination, the Inconel 617 face sheet must be made non-catalytic.

In this paper, a commercial ceramic coating has been evaluated as a non-catalytic coating for Inconel 617. Coated specimens were subjected to thermal shock cycles, surface emittances were measured, and surface equilibrium temperatures were measured for coated specimens and oxidized specimens exposed to an arc-tunnel environment.

TESTS

Both coated specimens and oxidized specimens of Inconel 617 were tested in this investigation. The latter specimens provided a basis of comparison for the coated specimen. The oxidized specimens were first cleaned until a water-sheeting surface was obtained, and then the specimens were oxidized for two hours at 1800°F in an atmospheric furnace. The resulting oxide was uniform and stable.

The ceramic coating is a commercially available, water base, silica-alumina type proprietary coating designated CRC-SBE.* The coating, which has been used extensively inside commercial furnaces to improve furnace efficiency was applied to the Inconel 617 specimens and cured at 500°F after application. Both 0.005 inch and 0.050 inch Inconel 617 specimens were coated. The thickness of the cured coating was 0.0015-0.002 inches.

Thermal Shock Tests

The thermal shock tests were conducted in an atmospheric oven. Sixteen test specimens of 0.005 inch Inconel 617, approximately 1.0 inches square and coated on one side, were used in the tests. The furnace was heated to 2000°F and held at that temperature. For a typical thermal shock cycle, the specimens were inserted into the hot furnace for 20 minutes and then taken out and allowed to cool at ambient conditions to near room temperature. Two

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* Produced by the Ceramic-Refractory Corporation, Transfer, PA. Use of trade names in this publication does not constitute endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

specimens were removed from the sequence after each ten cycles. Thus, the least exposed specimens were subjected to ten cycles, and the most exposed specimens were subjected to 80 cycles.

Emittance Measurements

The emittance of both oxidized and coated Inconel 617 specimens was measured using the apparatus and method described in reference 3. The emittance of the oxidized metal was measured after it had been oxidized in the furnace and after exposure in the arc-tunnel. The emittance of the coated metal was measured after curing and after 80 cycles of exposure in the furnace environment.

Arc-Tunnel Tests

Test models. - The configuration of the arc-tunnel test models is shown in figure 1. The test specimens, which were of 0.05 inch thick Inconel 617, were mounted on blocks of 50 lb/ft^3 fused silica using metal straps spot-welded about one inch from each corner of the specimen. The straps were inserted through holes in a silica block and fastened on the underside of the block. The surface of the specimen was about 1/8 inch below the surface of the copper wedge because previous tests (ref. 4) have shown that a more uniform heating distribution can be obtained with a rearward facing step than when the specimen is flush with the wedge. Four 30 gage chromel-alumel thermocouples were spot-welded symmetrically one-half inch from the center of each specimen. The thermocouple lead wires were covered with a silica cloth sheathing, inserted through a hole in the silica block, and taken out through a hole in the water-cooled copper wedge to a junction box.

Test Apparatus. - Figure 2 presents a simplified diagram of the Langley Research Center 1-Megawatt Aerothermal Arc-Tunnel. The arc-heated wind tunnel, which is described in reference 5, consists of a Thermal Dynamic F-5000 constricted-type d.c. arc-heater, a plenum chamber, a 15° conical supersonic nozzle with a 1.0-inch diameter minimum and a 6.0-inch diameter exit, a free-jet test section, a diffuser, and a 3-stage steam ejector. The arc-heater consists of a tungsten cathode and a water-cooled-copper anode. The arc is stabilized on a tungsten cathode with a nitrogen vortex. Oxygen can be added downstream to simulate an air mixture or additional nitrogen can be added for a 100% nitrogen flow. The arc is magnetically stabilized on the anode. The nominal stagnation pressures were 0.85 atm for simulated air and 0.75 atm for nitrogen.

Test Environment. - The test environments used in this investigation are given in Table 1. The local pressure at the test surface was measured with a calibration model the same size and shape as the test models. For the tests in air, the enthalpy was determined by using the following established facility procedure. Probes were used to determine the stagnation heating rate and stagnation pressure in the center of the stream. A correlation equation was then used to calculate the enthalpy using the theory of reference 6.

This established method could not be used to determine the enthalpy for the nitrogen test stream. Stray electrical current could be seen in the test chamber. Although none of this stray current appeared to attach to the test specimens, the heating rate probe was affected. Therefore, the enthalpy was determined using an energy balance method.

The baseline test environment was established by adjusting the arc-tunnel power settings until the oxidized specimen reached a temperature of approximately 1750°F when tested in air. The coated specimen was tested at the same power settings. For the tests in 100% nitrogen, the arc-tunnel current and mass flow rate were kept the same as for the tests in air. The resulting arc-tunnel voltage and specimen temperature were lower.

RESULTS AND DISCUSSION

Thermal Shock Tests

After the thermal shock cycles were completed, the specimens were examined using an electron microscope. Decarburization of the Inconel 617 was significant after as few as 10 cycles or 200 minutes at 2000°F (fig. 3). Decarburization is the main mechanism for the decrease in creep resistance of Inconel 617 at high temperature (ref. 7). Examination of the coated and uncoated sides indicates the coating did not affect the decarburization rate. Also, the coating appeared to be firmly attached to the metal even after 80 temperature cycles.

Emittance Measurements

The surface emittance of the specimens was determined by measuring the monochromatic normal emittance at several wavelengths between 1 and 15 microns and then integrating to obtain the total normal emittance at the test temperature.

The emittance of the oxidized surface was measured on two specimens. The emittance of one specimen was measured after being oxidized (fig. 4). The

other specimen was taken from the arc-tunnel specimen after it had been subjected to 4 cycles of arc-tunnel heating (fig. 5). The total emittance of both specimens was approximately the same, varying from about 0.77 at 990°F to about 0.82 at 1880°F.

The emittance of the coated metal was measured in the as received condition (fig. 6) and also measured with the specimen that was subjected to 80 thermal shock cycles (fig. 7). The total emittance of the as received coated specimen varied from about 0.70 at 990°F to about 0.65 at 1880°F. The total emittance of the 80 cycle specimen was about 0.79 at 990°F and about 0.75 at 1520°F. The emittance was inadvertantly not measured at 1880°F on the 80 cycle specimen, however the emittance would probably not change significantly from the 1520°F measurement (see fig. 6). These tests did not determine whether the increase in emissivity of the coated specimens after 80 thermal cycles was caused by the carbon-layer under the coating, which resulted from the decarburization of the metal, or by a change in the coating, which resulted from the long-time high temperature exposure.

Arc-Tunnel Tests

The results of the arc-tunnel tests are shown in Table 2 and figure 8. The maximum surface temperatures given in Table 2 are shown in figure 8 as the temperatures at 165 seconds when the surface had approximately reached equilibrium. The maximum surface temperature for each test and the measured emittance for each type of specimen were used to calculate a heating rate ratio for each test stream. The heating rate ratio is the emittance of the coated specimen (ϵ) times the fourth power of the maximum absolute temperature of that specimen (T) divided by the product of the same quantities for the

uncoated specimen. Inherent in these calculations is the assumption that the heat input to the specimens is equal to the heat output from the specimens which is approximately equal to the heat rejected by radiation. At maximum temperature conditions this approximation is satisfactory since the heat being stored and rejected by mechanisms other than radiation are insignificant compared to the heat rejected by radiation. Although a higher emittance for the coated surface would reduce the surface temperature and thus increase heat shield thermal efficiency, the heating rate ratio given in Table 2 would not change significantly even if the emittance were changed because the increase in ϵ would be countered by the decrease in T^4.

As can be seen from Table 2, the heating rate ratios are quite small--0.37 for the air stream and 0.29 for the nitrogen stream. These small ratios show that the coating is very non-catalytic compared to the oxidized surface.

The heating rate ratio is smaller in the nitrogen test stream than in air. Calculations of the stream composition using the equations of reference 8 and assuming chemical equilibrium indicated that in air essentially all the oxygen and 7 percent of the nitrogen was dissociated, where as in pure nitrogen, approximately 18 percent was dissociated. Because the catalytic effect is more pronounced in the nitrogen stream, these results indicate that nitrogen recombination rather than oxygen recombination was the dominant catalytic effect. Reference 9 suggests that at the surface temperatures obtained during these tests, the oxygen would recombine on both the coated and uncoated surfaces. Thus nitrogen recombination should be the dominant catalytic effect observed in these tests.

CONCLUDING REMARKS

A commercially available ceramic coating which is being considered for metallic thermal protection systems for advanced entry vehicles has been evaluated as a non-catalytic coating for Inconel 617. Coated Inconel 617 specimens were subjected to up to 80 thermal shock cycles, each cycle consisting of a 20 minute exposure in a 2000°F furnace. Microscopic examination showed that the coating did not interact with the metal and that the coating was still firmly attached to the metal after 80 cycles.

Surface emittance was measured on coated specimens and oxidized (2 hrs. at 1800°F in air) specimens. The measured emittance of the oxidized specimens varied from about 0.77 at 990°F to 0.82 at 1880°F. The emittance of the coated specimens varied from about 0.70 at 990°F to 0.65 at 1880°F.

The arc-tunnel test results showed that the coated surface was much less catalytic than the oxidized metal. In air, the heating rate on the coated surface was 37 percent of the heating rate on the oxidized surface and in nitrogen, the coated specimen heating rate was only 29 percent of the oxidized specimen heating rate. These results indicate that nitrogen recombination is the dominant effect in increased heating rates to catalytic surfaces.

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TABLE 1. - NOMINAL TEST CONDITIONS

	RECONSTITUTED AIR	NITROGEN
TOTAL PRESSURE, ATM.	0.85	0.76
TOTAL ENTHALPY, BTU/LB	5500	5000
MASS FLOW RATE, LB/SEC	0.044	0.047
MODEL STAGNATION PRESSURE, ATM	0.034	0.032
MODEL PRESSURE CENTER POINT, ATM	0.0015	0.0013
MACH NUMBER	4.1	4.2

TABLE 2. - ARC-TUNNEL TEST RESULTS

TEST STREAM	TEST SPECIMEN	ENTHALPY btu/lb	T_{max}, °F	HEATING RATE RATIO**
AIR	COATED	5590	1353	0.37
	UNCOATED*	5510	1753	
N_2	COATED	4890	1045	0.29
	UNCOATED*	4980	1495	

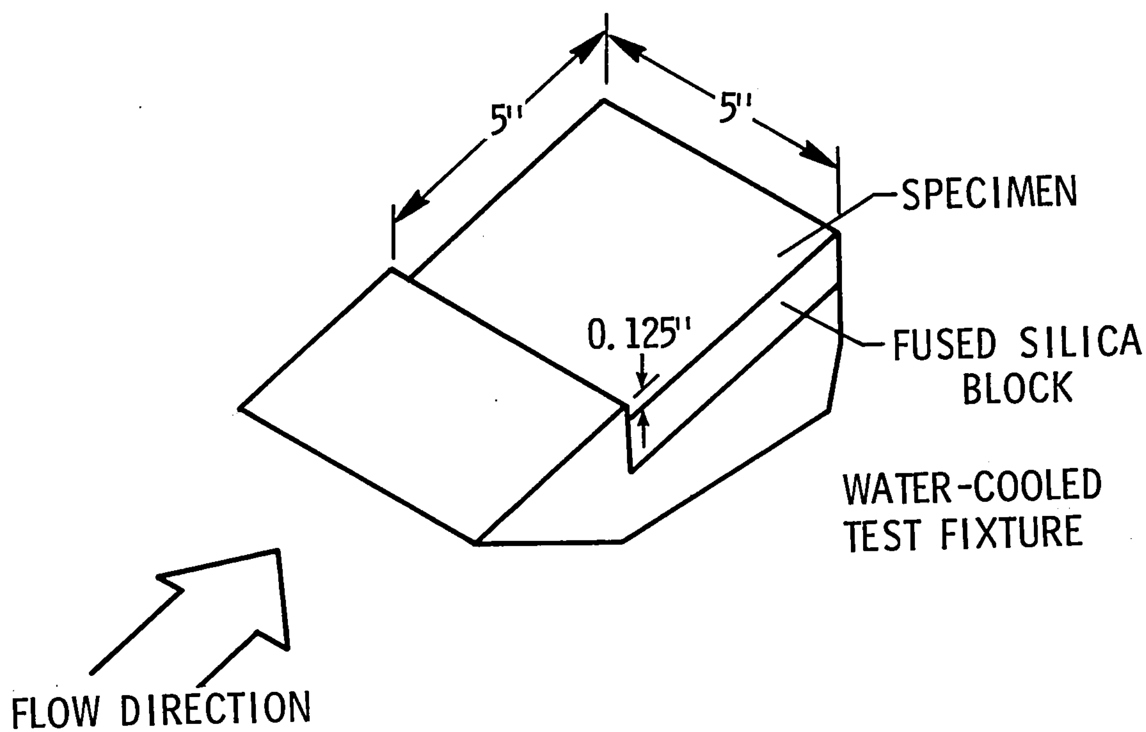

FIG. 1 - ARC-TUNNEL TEST SPECIMEN CONFIGURATION.

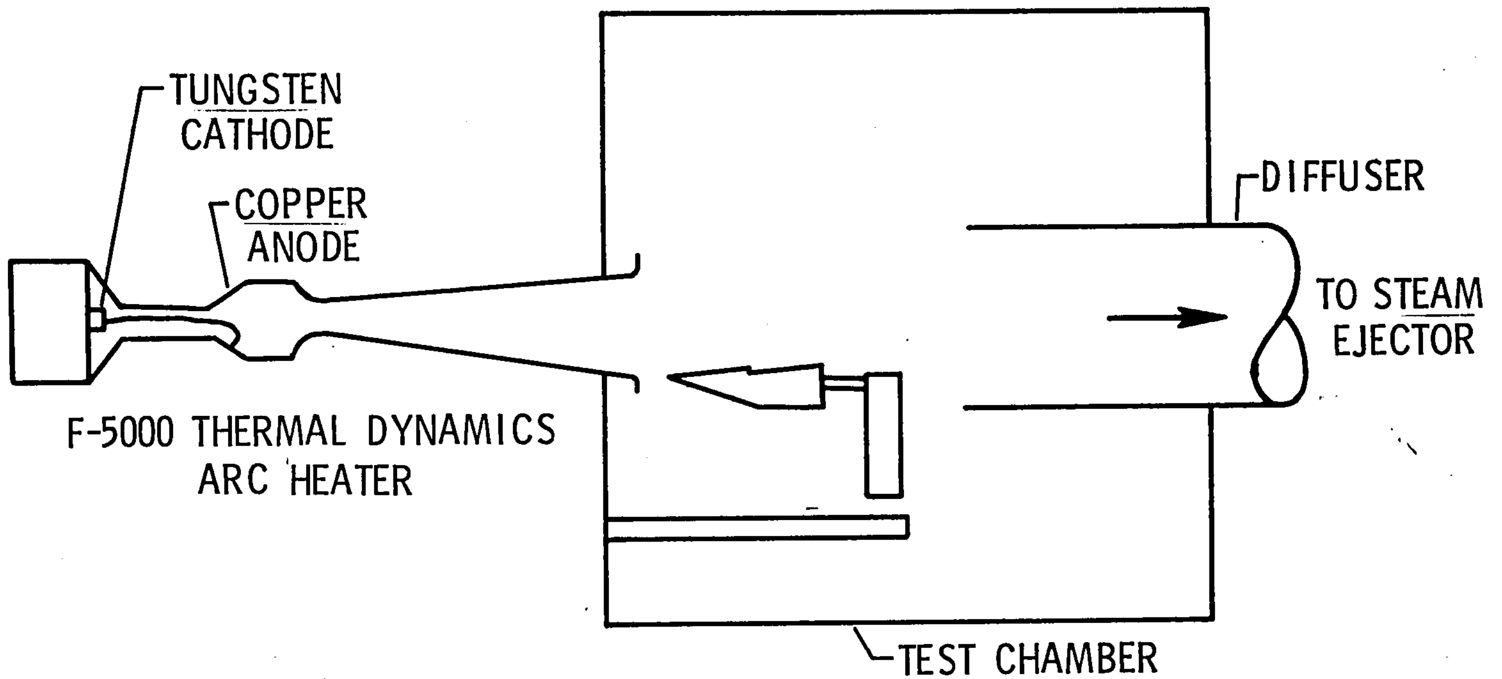

FIG. 2 - SCHEMATIC OF 1 MW ARC-TUNNEL.

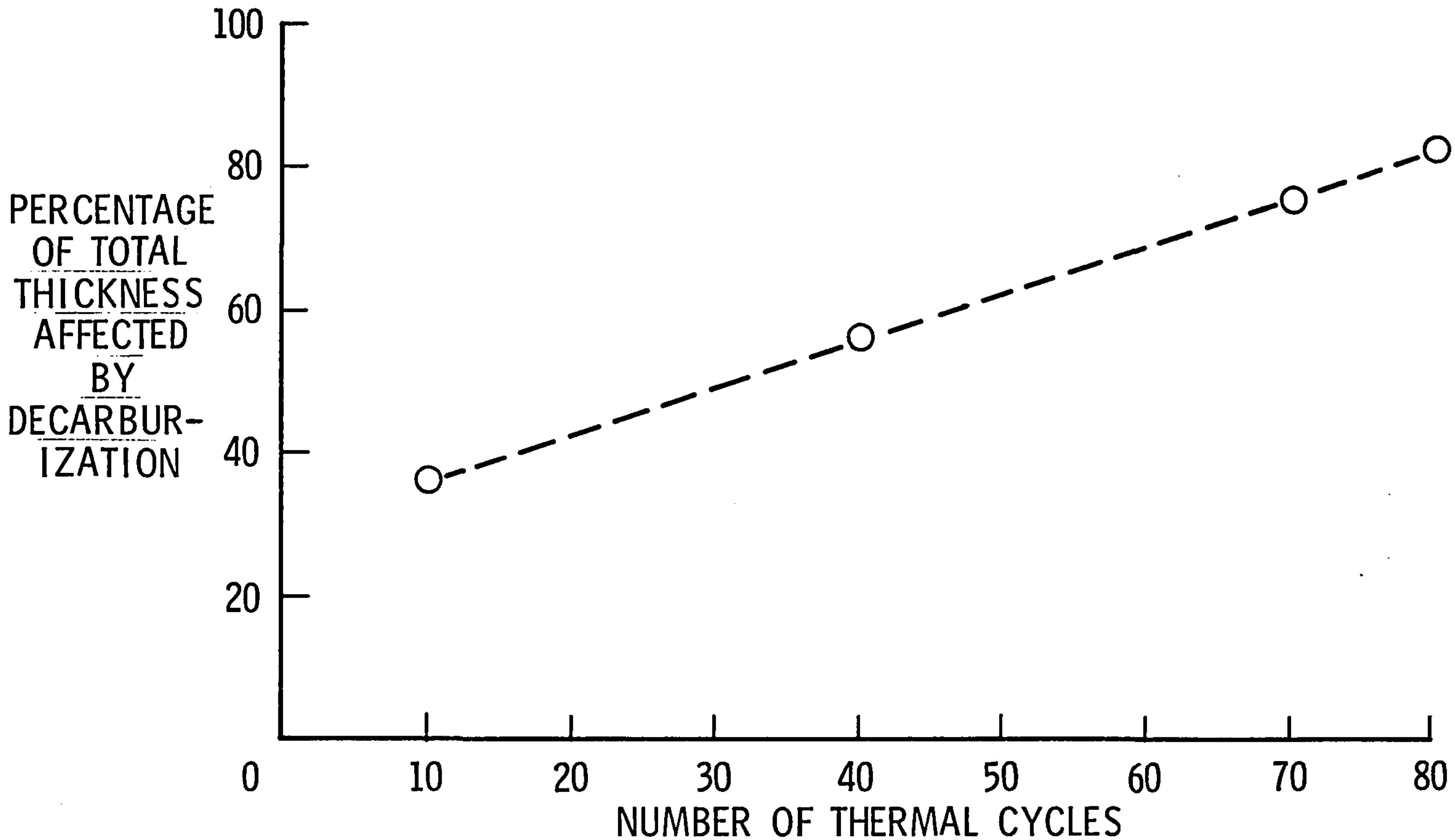

FIG. 3 - DECARBURIZATION OF INCONEL 617 BECAUSE OF THERMAL CYCLES.
(1 CYCLE = 20 MIN. AT 2000°F.)

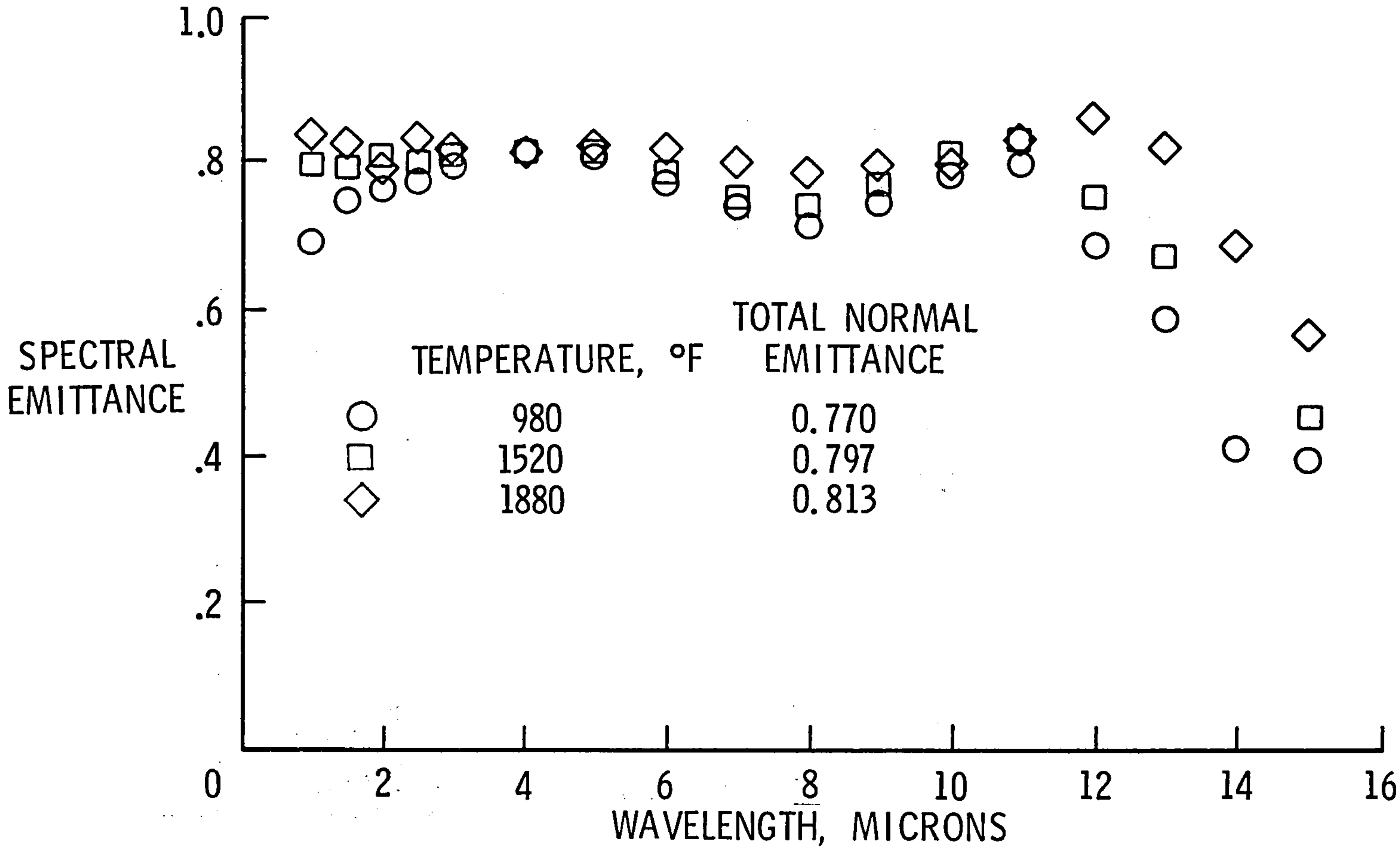

FIG. 4 - EMITTANCE OF OXIDIZED INCONEL 617.

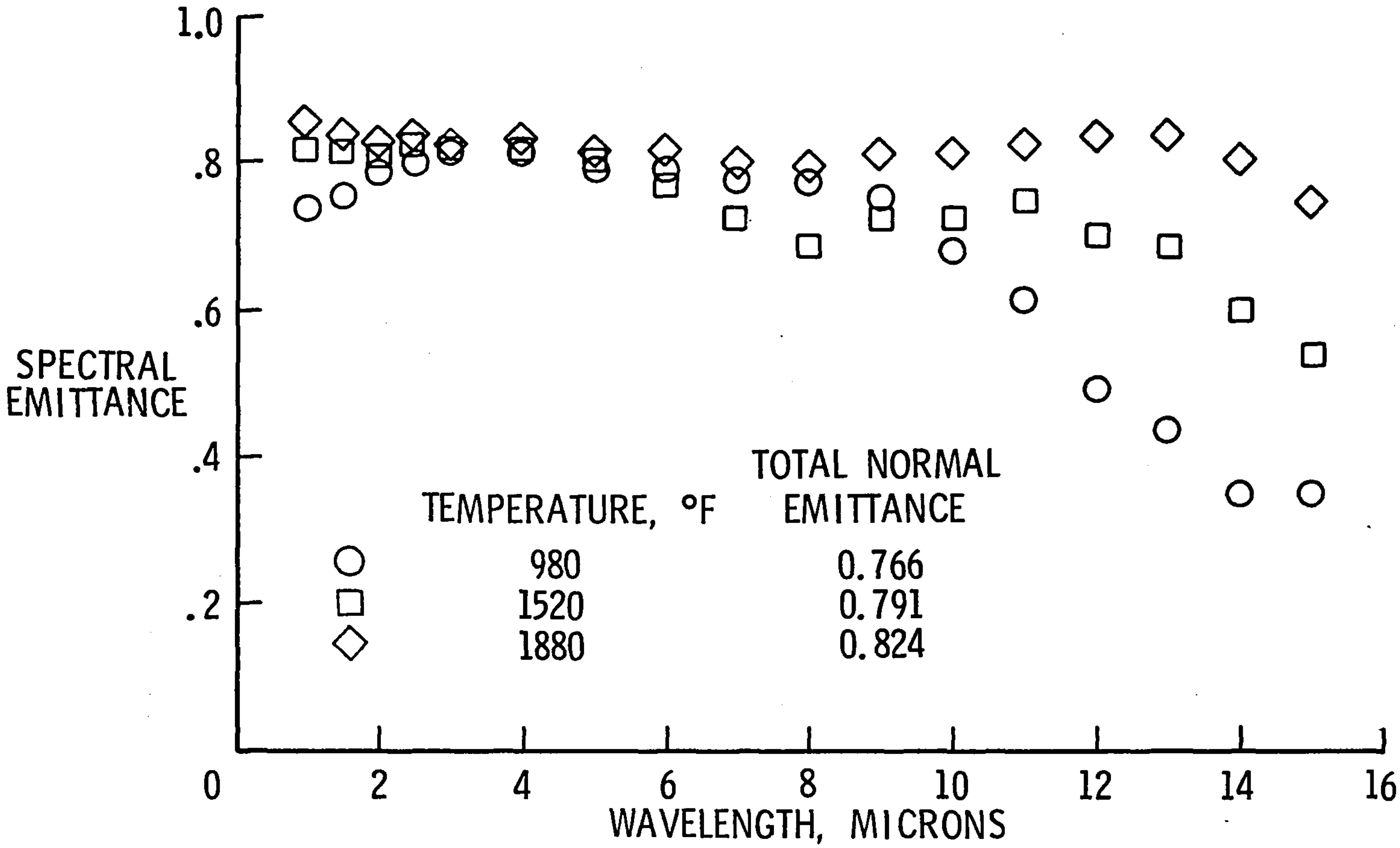

FIG. 5 - EMITTANCE OF OXIDIZED INCONEL 617 AFTER ARC-TUNNEL EXPOSURE.

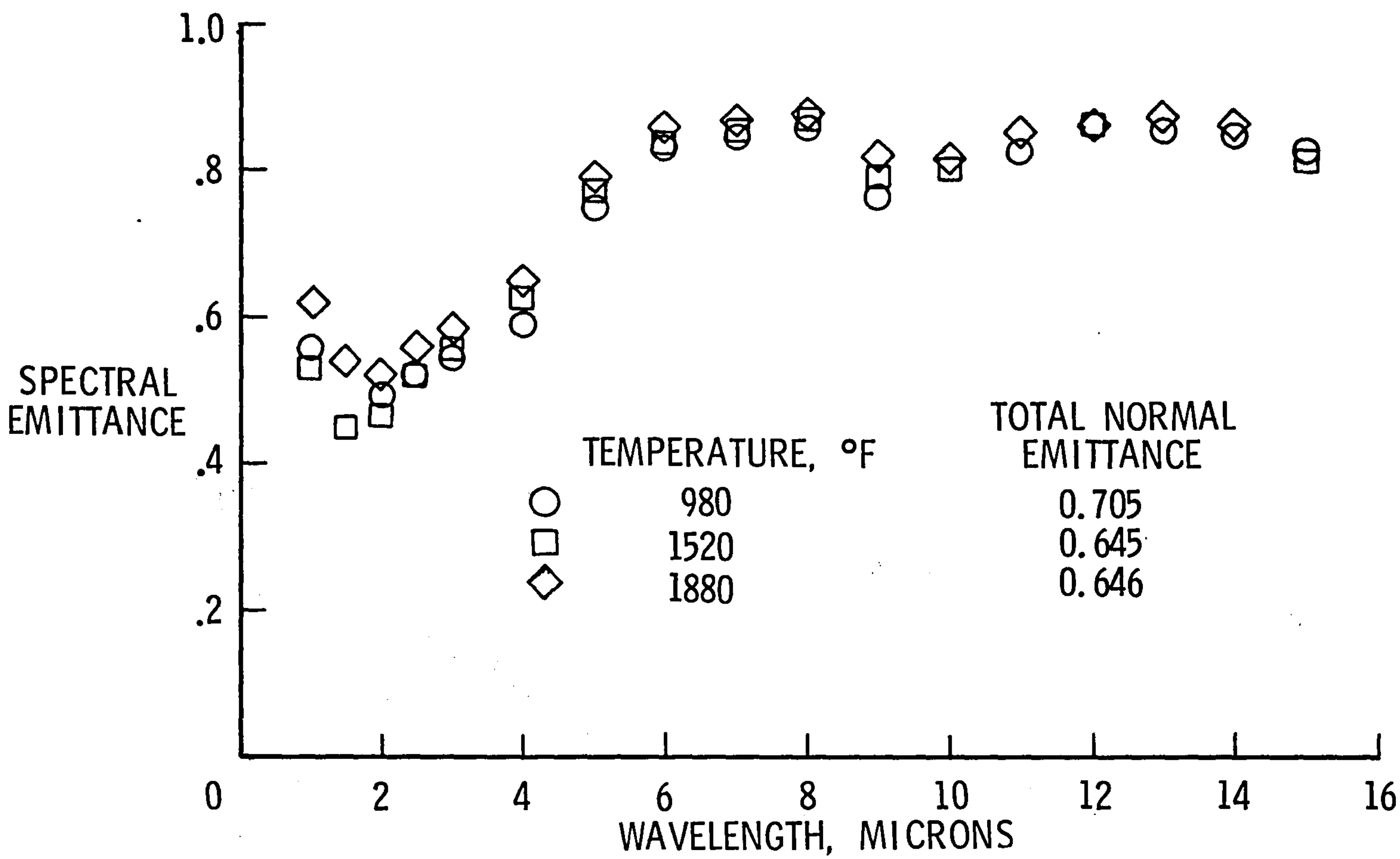

FIG. 6 - EMITTANCE OF COATED INCONEL 617.

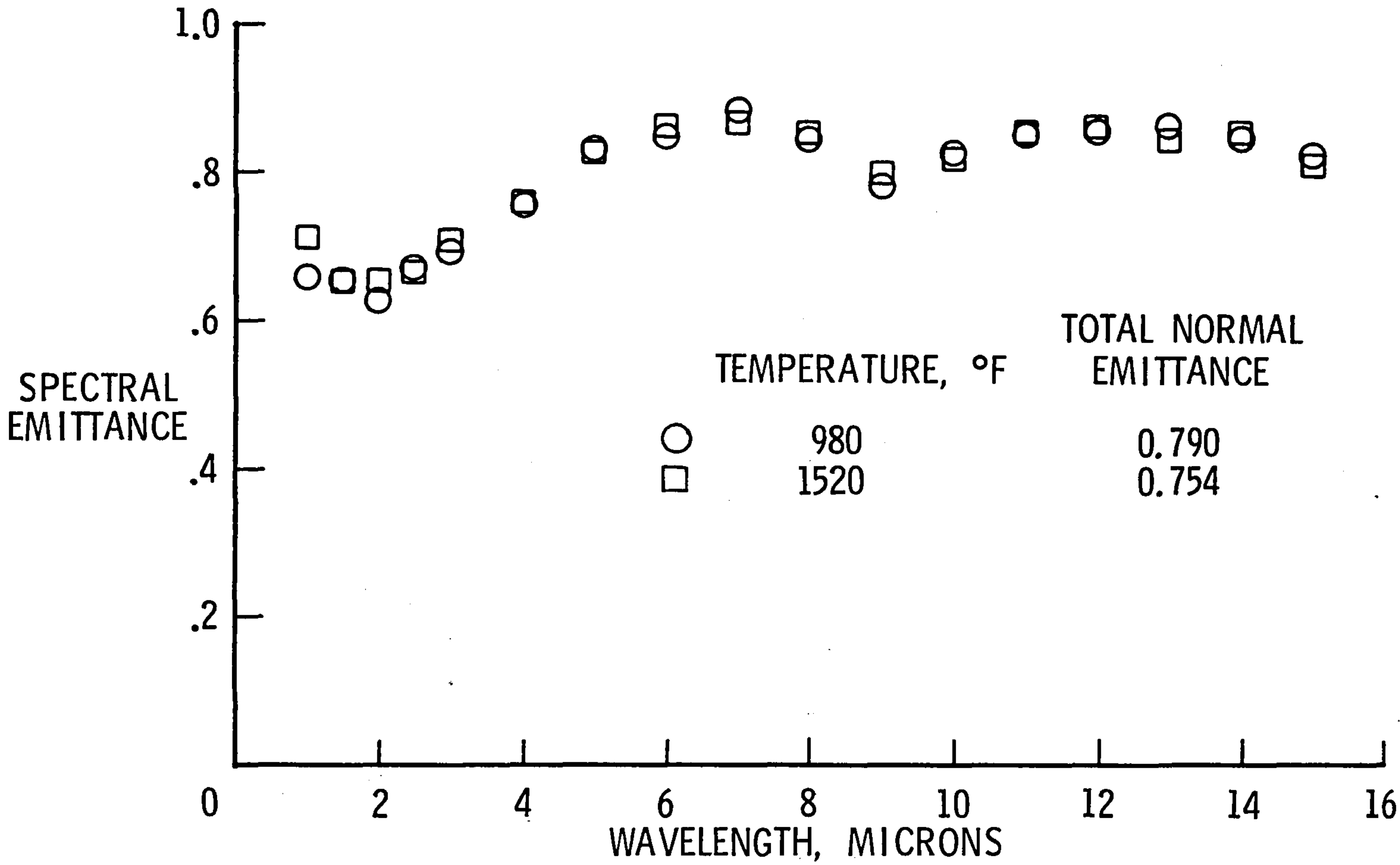

FIG. 7 - EMITTANCE OF COATED INCONEL 617 AFTER 80 THERMAL SHOCK CYCLES.

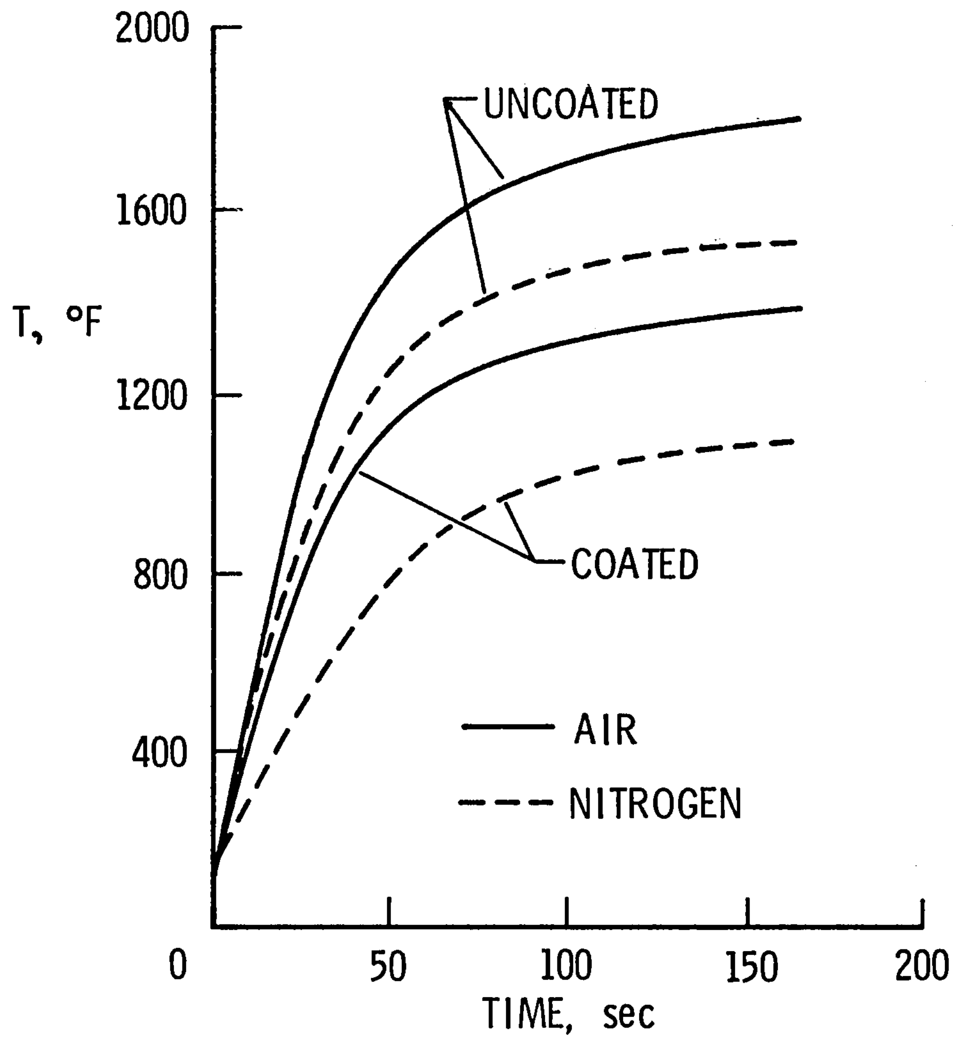

FIG. 8 - ARC-TUNNEL SPECIMEN TEMPERATURE HISTORIES.

1. Report No. NASA TM-85745	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Evaluation of a Non-Catalytic Coating for Metallic TPS		5. Report Date January 1984
		6. Performing Organization Code 506-53-33-04
7. Author(s) Claud M. Pittman, Ronald D. Brown, and John L. Shideler		8. Performing Organization Report No.
		10. Work Unit No.
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15. Supplementary Notes

16. Abstract

A commercially available ceramic coating has been evaluated for application to metallic heat shields for Shuttle-type entry vehicles. Coated Inconel 617 specimens were subjected to thermal shock cycles, surface emittances were measured, and surface equilibrium temperatures were measured for coated and oxidized specimens exposed to an arc-tunnel environment. The coating adhered very well to the metal and appeared to be very non-catalytic.

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